

POWDER METALLURGICAL PRODUCTION OF HIGH-TEMPERATURE-RESISTANT,
DISPERSION-HARDENED, TEMPERABLE NICKEL-BASE ALLOYS

K. H. Kramer

Translation of "Pulvermetallurgische Herstellung Hochwarmfester,
Dispersionsgehaerteter, Aushaertbarer Nickelbasislegierungen,"
Fried. Krupp G.m.b.H., Krupp Forschungsinstitut, Essen, 27 pages.

Report, 1973

(NASA-TT-F-15854) POWDER METALLURGICAL
PRODUCTION OF HIGH-TEMPERATURE-RESISTANT,
DISPERSION-HARDENED, TEMPERABLE NICKEL-BASE
ALLOYS (Techtran Corp.) 21 p HC \$4.25

N74-32964

Unclas
CSCL 11F G3/17 48572

STANDARD TITLE PAGE

1. Report No. NASA TT F-15854		2. Government Accession No.		3. Recipient's Catalog No.	
4. Title and Subtitle POWDER METALLURGICAL PRODUCTION OF HIGH-TEMPERATURE-RESISTANT, DISPERSION-HARDENED, TEMPERABLE NICKEL-BASE ALLOYS				5. Report Date September 1974	
				6. Performing Organization Code	
7. Author(s) K. H. Kramer				8. Performing Organization Report No.	
				10. Work Unit No.	
9. Performing Organization Name and Address Techtran Corporation P.O. Box 729, Glen Burnie, Md. 21061				11. Contract or Grant No. NASW-2485	
				13. Type of Report and Period Covered Translation	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D. C. 20546				14. Sponsoring Agency Code	
15. Supplementary Notes Translation of "Pulvermetallurgische Herstellung Hochwarmfester, Dispersionsgehaerteter, Aushaertbarer Nickelbasislegierungen," Fried. Krupp G.m.b.H., Krupp Forschungsinstitut, Essen, 27 pages. <i>Report 1973</i>					
16. Abstract The title objective is achieved by the mixing and milling of elemental powders or prealloys together with dispersate in an agitator ball mill (Attritor). The pros and cons of dry and wet milling, and choice of dispersate are discussed in detail. Charging coarse-grained intermetallic phases promotes milling to small dimensions; the fine-grained intermetallic phases promotes milling to small dimensions; the fine-grained powder absorbs less oxygen. Compaction of powders in an isostatic hot-press was optimum at 1200°C/1 hr/1000 bar. Microscopies showed good dispersate distribution. The fine-grained structure was superplastic at high temperatures.					
17. Key Words (Selected by Author(s))				18. Distribution Statement Unclassified - Unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified		21. No. of Pages 20	22. Price	

POWDER METALLURGICAL PRODUCTION OF HIGH-TEMPERATURE-RESISTANT, DISPERSION-HARDENED, TEMPERABLE NICKEL-BASE ALLOYS

K. H. Kramer

1. Introduction

High temperature-resistant superalloys have gained great importance in the last three decades for insertion in gas turbines. While some 15 years ago the weight quota of superalloys in gas turbines came to less than 20%, today the introduction of superalloys lies above 50% (1). Since raising the operating temperatures of heat engines, especially in aircraft turbines, leads to a considerable increase in their economy, a further development of superalloys is of special interest for still higher operational temperatures.

A survey of the temporal development of operational temperatures of high-heat-resistant materials for turbine driving buckets of aircraft turbines is shown in Figure 1.

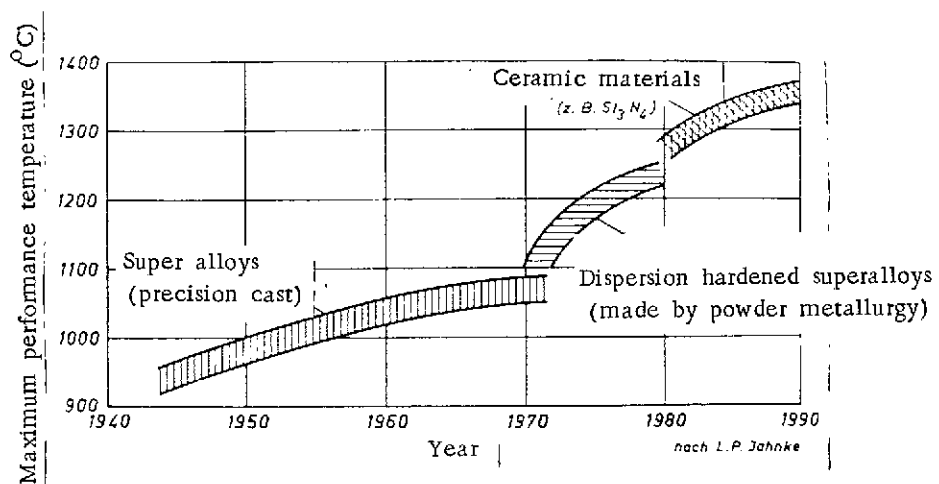


Figure 1. Development Over Decades of the Maximum Operating Temperatures of High-Temperature Resistant Materials for Turbine Buckets.

One can see from this that, according to present knowledge, the performance temperatures of superalloys, made by precision casting, asymptotically approach a 1050° C limit. In order to surmount this temperature limit, new kinds of

*Numbers in the margin indicate pagination in the foreign text.

industrial material groups, as e.g. dispersion-hardened superalloys or alloys of high-melting metals and the technologies necessary for their manufacture must be developed. The insertion of ceramic materials for temperatures above 1200° C is only to be expected considerably later.

The good temperature resistance of the best commercial superalloys is attained by crystalline solid solution-hardening and above all by the separation of the γ' -phase N_3 (Al, Ti).

This intermetallic phase, however, goes into solution at temperatures above /3 some 1000° C, with which process is linked a greater decrease in temperature resistance and above all in aging resistance. By still higher additions of the separation-hardening elements aluminum and titanium and the connected increase of the γ' portion in the alloy, the high temperature properties of superalloys indeed can still be improved slightly, but here limits are set, insofar as these alloys may no longer be prepared by smelting metallurgy owing too great liquification; besides, they are no longer forgeable.

Since the mechanism of separation hardening is effective up to temperatures of about 1000° C, (the particles coagulate and are completely dissolved in the matrix at still higher temperatures), an improvement of the heat-resistance and time-conditioned properties of superalloys can be achieved only by introduction of other hardening mechanisms. Dispersion hardening offers this. By that - mostly by the processes of powder metallurgy - particles of hard matter, e.g. oxides, nitrides, carbides, etc. become embedded in the matrix, which (particles) are as inert as possible in respect to the matrix up to temperatures near the melting point and thus do not interact with the matrix at all.

The action of mixed-crystal and separation hardening in superalloys in comparison to dispersion hardening of pure nickel is plain from Figure 2. While the superalloys exhibit a marked drop in time-stability at very high temperatures, the dispersion-hardened TD-nickel shows only a slight drop in strength. A combination of the hardening mechanisms here leads to a new alloy type, to the dispersion-hardened, temperable superalloys. Although this direction of development already had been evident for years, yet "mechanical alloy" was first successful in 1970 (2) by means of a new powder-metallurgical technology, in /4

developing a dispersion-hardened temperable superalloy, the time-stability of which in the temperature range above 1000° C lay above both the commercial superalloys and the TD-nickel. The goal of the present experimental work is a consistent further development of this new kind of powder metallurgical manufacturing process and with it additional improvement of the high temperature properties of superalloys up to operational temperatures of about 1200° C.

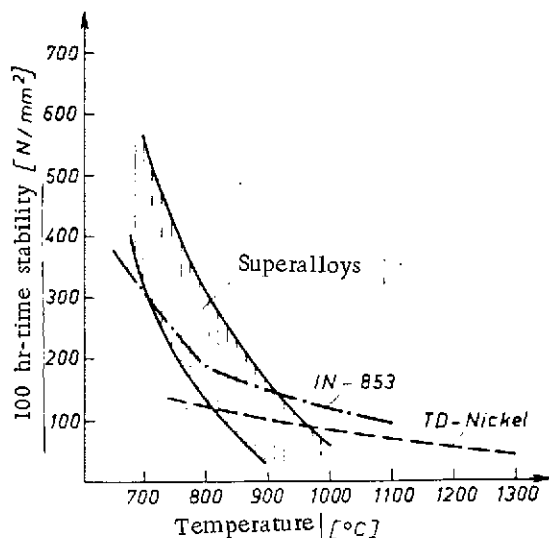


Figure 2. Temperature Function of the 1000 Hour-Time Stability of Superalloys, ID-Nickel and IN-853.

In comparison it is more advantageous to mix and mill in ball mills. The most effective particle breakdown of metal powder particles to "submicron" powder, as a requirement for an efficacious dispersion hardening, has been reached only in a Ruhr Works ball mill¹.

In B) The internal oxidation is mostly limited to binary systems, since, in higher alloyed materials, the diffusion rate of oxygen in the matrix is too slight. In this case a covering layer is formed, which can hinder extensively an inner oxidation.

2. Processes For Production of Dispersion-Hardened Industrial Materials

In Table 1 are four ways of processing, fundamentally different from one another, for the manufacture of dispersion-hardened materials: A, mechanical mixing; B, oxidation; C, chemical precipitation; D, electrochemical deposition. /5

In A) By the exclusive mixing of metal or alloy powders with a dispersate, no good dispersate distribution can set in because of lack of particle comminution.

¹Attritor - registered trademark.

In C) Probably the best-known process for the production of dispersion-hardened nickel (TD-nickel) is the chemical precipitation of a nickel salt in the suspension of dispersate. A further development of this process in respect to a general precipitation of several metal salts has already been successful. Materials prepared by these processes show an outstanding dispersate distribution, yet these processes are extremely useful.

In D) The electrochemical deposition of metals in a suspension of the dispersate can be used in the manufacture of dispersion-hardened pure metals. Transfer of this method of operation to the production of alloys, according to results to date, is not promising.

TABLE 1. PROCESSING ROUTES FOR PRODUCING DISPERSION-HARDENED MATERIALS.

A	B	C	D
Mechanical Mixing	Oxidation	Chemical Precipitation	Electrochemical Deposition
Mechanical Mixing of metal or alloy powders and dispersate.	Internal oxydation of alloy powders.	Chemical precipitation of a metal salt in the suspension of the dispersate (TD-nickel).	Electro-chemical deposition of metal in a suspension of the dispersate.
Mixing and milling of metal powders and dispersate in the attritor.	Surface oxydation of metal powders (SAP-aluminum).	Common precipitation of several metal salts in the suspension of the dispersate.	
Mixing and milling of metal powders and dispersate in the attritor and production of the metal (T. N.: word illegible).	Oxydation of alloy powders with subsequent selective reduction.		

Besides the production processes named there still are a number of others (e.g. freeze drying), which will not be examined closer here.

In the choice of a suitable process for production of dispersion-hardened, temperable nickel alloys, it has to be considered that the alloys must contain the separation hardening elements, e.g. aluminum and titanium, i.e. it eliminates all those processes in which the powder becomes converted into highly oxidized form and must subsequently be reduced. Since aluminum and titanium oxides possess such high enthalpies of formation that a reduction in hydrogen is not possible, as production technology, the mixing and milling of metal powders

together with the dispersate in the Attritor under complete exclusion of air is offered. In America some successful researches for production of dispersion-hardened nickel alloys have been conducted with the help of the Attritor (1 - 15). These are the different techniques used: Wet milling (3 - 9) with a milling liquid, e.g. alcohol or heptane + oleic acid, and dry milling (2, 10 - 16) without milling additives at all, which is known by the name "mechanical alloy". In the first procedure the metal powder particles are milled individually to grain sizes under 1 μm ; in the second the elementary powders become bonded together to a powder aggregate with particle sizes of about 200 μm . /7

The production methods of chemical precipitation, which compared with other processes, occur far more frequently in patent literature and, as known, lead to exceptional distribution of the dispersate, are unsuitable as processes for the production of dispersion-hardened, temperable nickel alloys, since in all cases oxide phases are formed, which, in the instances of aluminum and titanium, cannot be reduced again.

3. Choice of a Suitable Dispersate

In the choice of a useful dispersate for dispersion-hardening of nickel alloys, two quantities play the decisive role: The finely divided dispersate in the matrix must possess high stability both thermodynamically and thermally. For thermodynamic considerations therefore, primarily the oxides with high free formation enthalpies come into question. For a better view, in Figure 3 the free formation enthalpies of some stable oxides as a function of temperature are plotted. The ThO_2 used in the dispersion hardening of TD-nickel next to CaO has the highest enthalpy of formation. However it is radioactive, even though weakly so, hence its inclusion should be avoided as far as possible. Therefore in recent years yttrium oxide has been preferred as a dispersate in nickel alloys.

In the present studies Al_2O_3 , MgO , Ce_2O_3 , La_2O_3 and Y_2O_3 were used as dispersate. /8

It has been shown, however, that MgO , whose free formation enthalpy of which lies partly even above that of Y_2O_3 , or Al_2O_3 at annealing temperatures above 1200° C, are not stable and a marked coagulation of the particles in the alloy

appears, which doubtless lessens the heat resistance. Coagulation processes are influenced by the boundary surface energy between dispersate and matrix. At a high energy the dispersate tends to coagulate. If one can lower it e.g. by /9 changed alloy compositions, (17), then one increases by it also the thermal stability of the dispersion-hardened alloy.

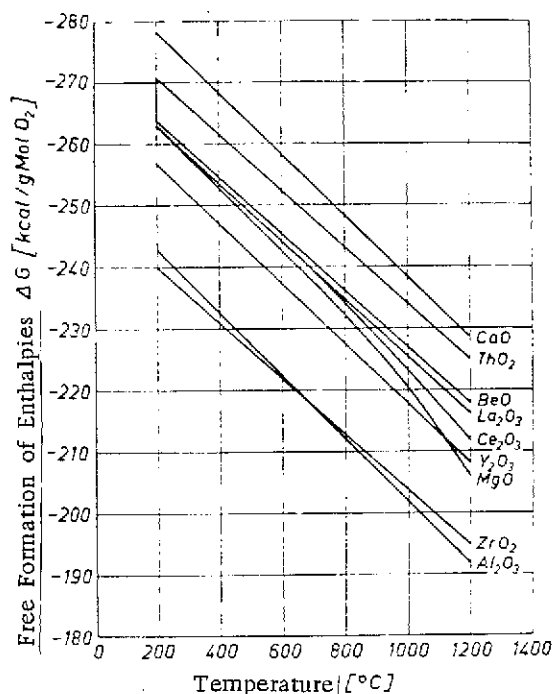


Figure 3. Free Enthalpies of Formation of Oxides (Calculated After Data From: Handbook of Chemistry and Physics 1969/70).

An additional important choice criterion for the dispersate is its price. Yttrium oxide at a price of about 500 Dm/kg is a quite expensive dispersate. Very unfavorable in cost are most of the rare earth oxides, with exception of Ce_2O_3 and La_2O_3 , which are far cheaper than Y_2O_3 . Accordingly a series of researches with these dispersates also was done. It showed that their stabilities up to temperatures of some 1300° C are to be equated with that of Y_2O_3 .

4. Experimental Execution and Results

4.1 Milling Tests in the Attritor

Two Attritors with a capacity of about 5 liters were available.

Steel or hard metal carbide balls with 6-8 mm Ø were used. The Attritor receptacle and stirrer were protected against too strong abrasion by a nickel-based wear-resistant alloy, and the containers were closed vacuum-tight, so as to hinder oxygen absorption by the metal powder during milling.

The starting powders for milling are summarized in Table 2. Both elemental powder and prealloys were used. By inserting of coarse-grained intermetallic phases, on one hand a good milling was obtained, on the other hand the oxygen content of the powder mixture was kept low.

TABLE 2. STARTING POWDER FOR MILLING IN THE ATTRITOR
(PURE METALS AND PREALLOYS).

Metal or Prealloy	Grain size (um)	Oxygen content (%)
Ni	4 - 7	0,10
Cr	150 - 350	0,08
Co	2-4 / 5-10	0,49 / 0,03
Mo	2,5 - 5	0,15
Ni-30Al	1000	0,02
Ni-20Al-20Ti	1000	0,16
Mo-22,8Al	1000	0,10
Co-57Cr	1000	0,03
Co-60Mo	1000	0,09

Commas indicate decimal points.

The alloy compositions of the powder mixtures correspond to those of modern /10
superalloys with a dispersate addition of 2.5% by volume. Most of the investi-
gations were conducted on alloys of the following estimated composition:

I.) Ni Cr[sic]15 Co18.5 Mo5.2 Al 4.25 Ti[sic]3.5 + 2.5%/volume Y_2O_3

(Δ Udimet 700 + dispersate) and

II.) Ni Cr 16 Co 8.5 W 2.6 Mo 1.75 Ta 1.75 Nb 0.9 Al 3.4 Ti 3.4 + 2.5%/volume
 Y_2O_3 (Δ IN 738 + dispersate).

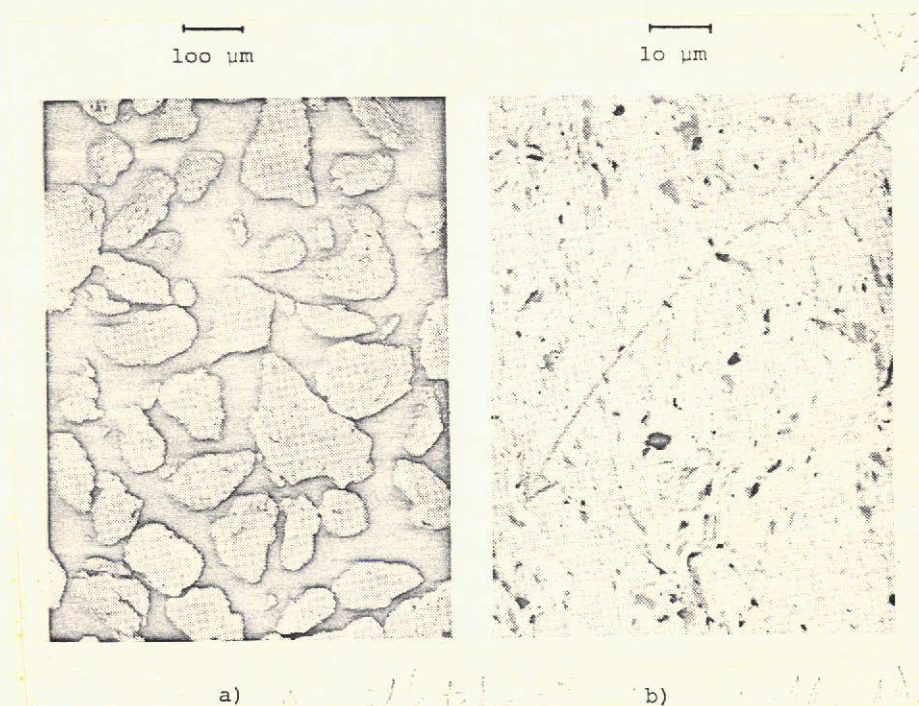
4.1.1. Dry Milling

With dry milling, in the "mechanical alloy" the metal powder mixture and /11
dispersate were filled together into the Attritor under flowing argon and, under
vacuum, were milled 20 - 40 hours at 140 rpm. Thus, from the charged fine-
grained elemental metal powders and the dispersate, there arose a powder
agglomerate with a grain size of about 100 - 200 um, as Figure 4a shows. At a
greater enlargement (Figure 4b), the various metal powder particles are still
recognizable, while the dispersate is not visible owing to its fineness. The
powder mixtures made in this way were separated from the balls by sieving and
then hot-compressed.

4.1.2. Wet Milling

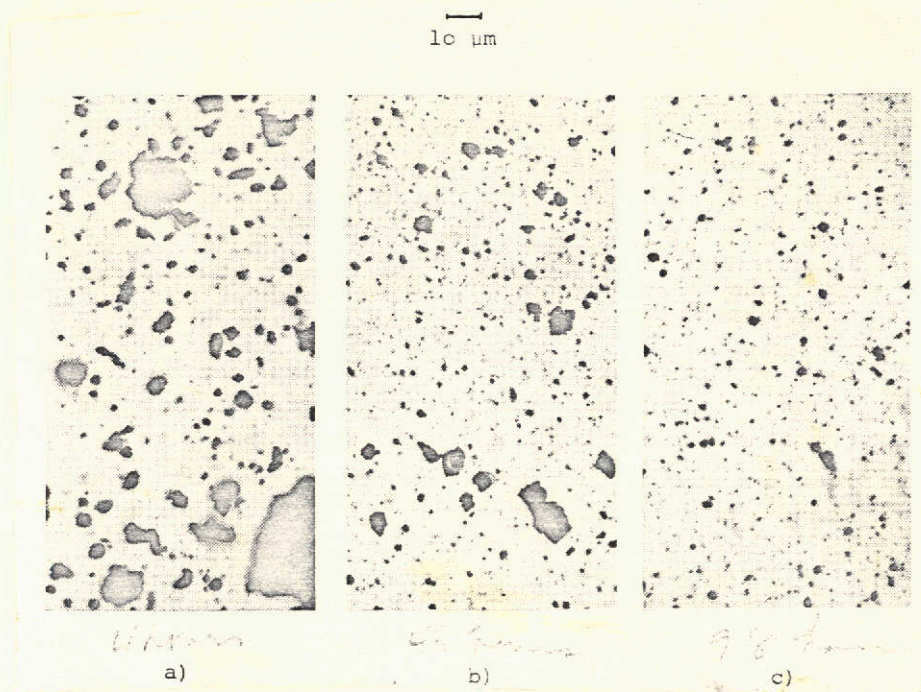
In wet milling metal powder mixtures and dispersate were milled 30 - 100 /12
hours in a grinding liquid, partially with addition of milling aids. At times

during the milling as much milling liquid was added (as needed) to make the slurry still slide well off a spatula. Various milling liquids (e.g. heptane, ethanol, dichloromethane, etc.) and aids (e.g. oleic acid, stearic acid) were used. According to the present experiments, non-polar milling liquids like hexane or heptane, because of their poor milling behavior, are only conditionally useful. By addition of oleic acid, however, the milling effect can be improved. Far more suitable for milling fluids are methanol or dichloromethane with addition of yttrium nitrate, whereas, by an associated glowing, the nitrate becomes decomposed to the oxide. The milling results with dichloromethane and yttrium nitrate for 4, 48 and 98 hours milling time are reproduced in Figures 5a-c. The finest powder portion after 98 hours in comparison to other millings is relatively high. But the inclusion of yttrium nitrate turns out to be a disadvantage, since an oxidation of the metal powder mixture is favored by the crystal water content and the decomposition of the nitrate upon glowing. Even with the use of pre-purified, practically anhydrous methanol or acetone (with molecular sieve), a considerable uptake of oxygen still appeared through the partial decomposition of the milling liquid.



Figures 4a-b. Dry-Milled Powder Mixtures of the Composition Ni Cr15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5%/volume Y_2O_3 (unetched).

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Figures 5a-c. Wet Milled Powder Mixture of the Composition
Ni Cr[sic]15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5%/volumd Y_2O_3 ;
Milling Liquid: Dichloromethane; Milling Aid: Yttrium Nitrate.

After milling, the slurry was filled into a container through a bottom valve under vacuum. Then the liquid was distilled off in a rotary tube furnace, and the powder glowed at temperatures up to $1150^{\circ}C$ under purified hydrogen with a dew point below $-75^{\circ}C$. Upon glowing, the powder sintered together and thereafter was no longer pyrophoric and so is easy to handle.

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4.2. Experiments to Compact the Powder

4.2.1. Hot Extrusion

While the dry milled powder mixtures showed a space filling of about 50% and could be directly encapsulated or extruded without pre-pressing, the wet milled powder with space fillings of 30 - 50% was previously compressed cold at a pressure of 3600 bar isostatically. After that the space fillings measured 60 - 68%. The powder or pressings were heat-sealed in steel capsules of 56 mm outer diameter, evacuated and extruded to 17-mm rods at $1150^{\circ}C$ with a maximum press force of 3000 MN and a compression ratio of 10 : 1.

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4.2.2. Isostatic Hot-Compression

The majority of the powder prepared by dry- and wet-milling was compressed in an isostatic hot press (ASEA). Cylindrical capsules from 0.5 mm thick iron tubing of 17 mm diameter were used. The dry-milled powder was loosely shaken in, while the wet-milled powder, owing to its low filling density, was carefully pressed into the capsules with a crank press. The capsules were closed at a temperature of 500° C and a vacuum of 10^{-5} Torr ($\approx 1.3 \times 10^{-2}$ ubar) and were inserted in the isostatic hot-press. Table 3 gives a short review of the pressing conditions, and the respective results of the compression. At a pressing temperature of 1150° C, even at the highest pressure of 3200 bar, an incomplete compaction was attained. At a temperature of 1200° C, a pressure of at least 1000 bar has to be applied in order to get dense samples. At the highest temperature of 1250° C, even a pressure of 500 bar suffices for complete compaction. Figure 6 shows some capsules after compression in the isostatic hot press. The iron casing has partially developed since at less than the 50% space filling of the charged powder was very small.

TABLE 3. PRESSING CONDITIONS AND RESULTS IN ISOSTATIC HOT PRESSING.

/15

Pressing temperature, [°C]	Time (hrs)	Pressure (bar)	Results
1150	1	500	Not completely compressed (pores)
1150	1	1000	Not completely compressed (pores)
1150	1	3200	Not completely compressed (fine pores)
1200	1	500	Not completely compressed (fine pores)
1200	1	1000	Dense
1200	1	3200	Dense
1250	1	500	Dense
1250	1	1000	Dense
1250	1	3200	Dense

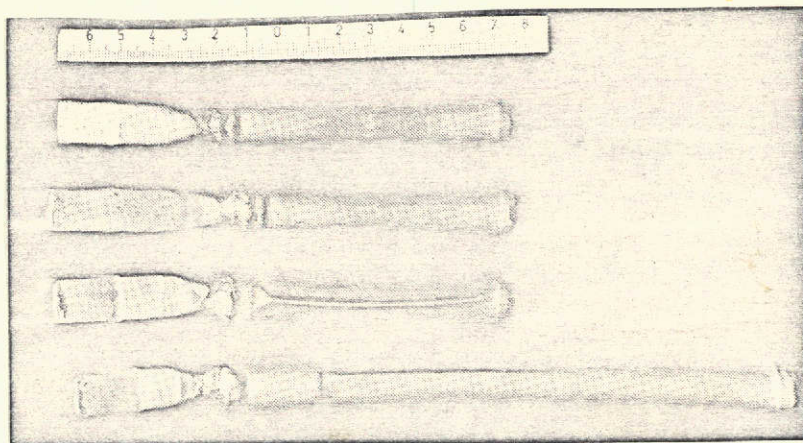


Figure 6. Isostatic Hot-Pressed Capsules, Pressing Conditions: 1200° C/1 hour/1000 Bar.

4.3. Metallographic Studies on Compressed Powders

Some typical pictures of isostatic hot pressed samples were selected which /16 characterize the distribution and formation of the dispersate.

Figure 7 shows a dry milled powder isostatically hot pressed at 1200° C. The dispersate lies finely divided in the matrix. To be sure, isolated dispersate-free inclusions appear. Here it is a case of coarse metal powder particles which were not milled finely enough. The lightly etched sample allows no recognition of grain boundaries, indicating that an extremely fine-grained material was present. This assumption was confirmed by electron microscopic pictures. By isostatic hot-pressing at some 1250° and 3200 bar, a marked grain growth was attained, as can be seen from Figure 8. Thus grain sizes above 100 μm appeared.

By wet-milling with acetone or methanol, an extremely fine "submicron" /17 powder was prepared, which after compression in the isostatic hot press shows a good oxide distribution (Figure 9), although the oxide proportion in the sample still is too high. By the use of a chlorinated milling liquid, e.g. carbon tetrachloride, the oxygen content of the powder indeed was lowered by glowing under hydrogen, but the homogeneous oxide distribution was lost in the process (Figure 10).

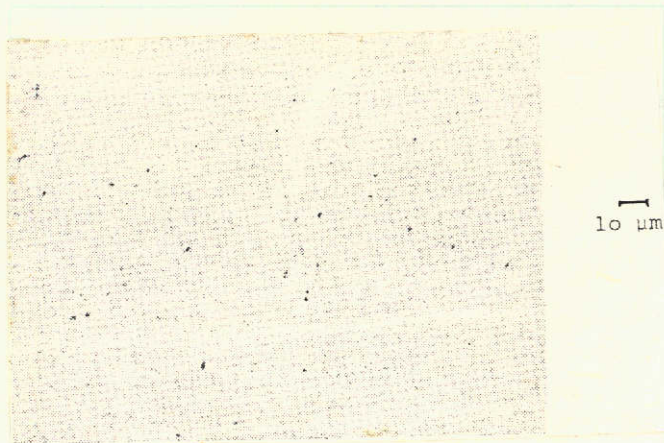


Figure 7. Dry Milled Powder Mixture of the Composition Ni Cr15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5%/volume Y_2O_3 , Isostatically Hot-Pressed: 1200°C/1 hr/1000 Bar (Lightly Etched).



Figure 8. Dry Milled Powder Mixture of the Composition Ni Cr15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5%/volume Y_2O_3 , Isostatically Hot-Pressed: 1250°C/1 hr/3200 Bar (Polished).



Figure 9. Powder Mixture Wet-Milled With Acetone; Composition Ni Cr15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5% Volume Y_2O_3 , Isostatically Hot-Pressed: 1200°C/1 hr/1000 Bar (Polished).



Figure 10. Powder Mixture Wet-Milled With Carbon Tetrachloride; Composition Ni Cr15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5% Volume Y_2O_3 , Isostatically Hot-Pressed: 1200°C/1 hr/1000 Bar (Polished).

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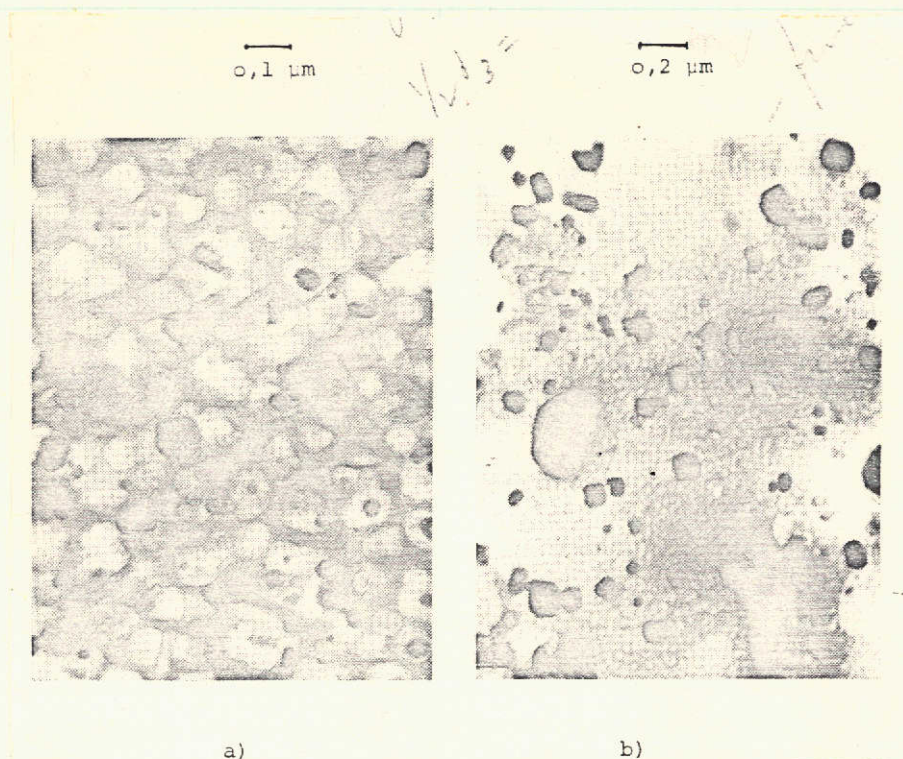
4.4. Electronmicroscopic Studies

The electron microscope pictures of powders compressed by isostatic hot-pressing supplement the metallographic examinations, since with their help, the particle form and distribution of the dispersate are captured more accurately, and also, with temperable alloys, the separated Y'-particles in general could be made visible first. Figure 11a shows a dispersion-hardened, temperable nickel alloy in the solution-annealed state, which had been produced by dry milling and isostatic hot-pressing. With the 1270° C glowing and associated slow air-cooling, relatively coarse Y'-precipitates are formed (bright particles of about 0.1 μ m grain size), which have about the same size as the dispersate (black particles). After an additional heat-treatment to temper the alloy, additional fine Y'-particles have been deposited (Figure 11b), which then are around 0.01 μ m in size and are presented beside the electrolytic thin, dissolved-out, coarse, primary Y'-phase. The greater etching out of the Y'-phase by the use of sulfuric acid electrolytes has the advantage that a distinct difference between Y'-particles (bright) and dispersate (dark) is possible, as Figures 11a-b show. /19

5. Discussion of the Experimental Results

Research has shown that it is possible to produce dispersion-hardened, temperable nickel alloys by dry or wet milling in the Attritor and associated compaction by hot extrusion or isostatic hot pressing. The two different production processes for the powder mixtures demonstrate advantages and disadvantages, which are discussed first:

The dry-milling process developed by J. S. Benjamin (2) offers the advantage that the charged powder, during milling in the Attritor, comes into contact with neither milling liquid nor milling aid. Therefore contamination during milling can not occur. Since the grinding balls, agitator, and vessel walls are coated with a packed powder layer, which is partially detached and again built up, contamination of the powder mixture by abrasion likewise is excluded. But it works out to disadvantage that coarse grained powder charged (e.g. Cr) in part doesn't get milled finely enough, so that, in the compacted alloy, coarse inclusions appear (Figure 7), containing no dispersate. Here a remedy can be use of more brittle intermetallic phases, more easily comminuted by the mill and which therefore may be inserted in coarser form with lower O₂ content. /21



Figures 11a-b. Dry-Milled Powder Mixtures of the Composition Ni Cr15 Co18.5 Mo5.2 Al 4.25 Ti 3.5 + 2.5%/volume Y_2O_3 , Isostatic Hot Pressed: 1200°C/1 hr/1000 Bar (Electron-microscopic Irradiation). 11a: + 1270°C/2 hrs/air, coarse Y'-deposition (bright) and Y_2O_3 particles (dark). 11b: + 1270°C/2 hrs/air + 1080°C/5 hrs/air + 800°C/15 hrs/air; dissolved out, coarse Y'-depositions, fine Y'-precipitates (bright) and Y_2O_3 particles (dark).

After milling, the necessary separation of balls and powder by sieving risks coating the active powder surfaces with an oxide layer which cannot be eliminated later. The oxygen absorption can be illustrated by the milled powder which is not yet being homogenized, as Figure 4b has shown. The powder agglomerate contains the charged metal powder particles still in the elemental form, and in this process, those with a high formation enthalpy of the oxide absorb oxygen especially readily. The electron microscope pictures (Figures 11a-b) show a good distribution of the dispersate. The oxide particles occur separated from one another, yet the particles at some 0.1 μm are somewhat coarser than in dispersion-hardened alloys made by chemical processes (e.g. TD-nickel). It remains to be investigated whether charging a finer dispersate will yield finer grain size.

Much has been written on wet milling of powders in ball mills or Attritors. In some works, the milling behavior of metal powders as a function of milling liquids and aids is studied, (18 - 20). These, however, very seldom are for use of powder mixtures containing aluminum and titanium, since these elements with their high affinity for oxygen are easily oxidized. The experiments have shown that the usual milling liquids like acetone or methanol lead to a good pulverization, but, on the other hand, a considerable oxidation of the metal powder occurs. The problem in wet milling then is to use a milling liquid which provides good milling without entering into any reaction with the metal powder mixture. In the use of acetone, as Figure 9 shows, a homogeneous distribution of the dispersate is attained. In contrast to dry milling, coarse powder particles in the form of dispersate-free inclusions appear. However, the oxygen content of the alloy still has to be lowered. The use of pure hydrocarbons as hexane, heptane, etc. is excluded because of poor milling results, as are the chlorinated liquids because of their reaction with the metal powders. The milling action of hexane or heptane, however, can be improved by additives like oleic acid, stearic acid or ethanol, so that these practically anhydrous liquids can surely be used for milling. /22

The wet milling process then has gained significance in the manufacture of dispersion-hardened, temperable alloys when one is successful in finding a suitable milling liquid which fulfills both requirements - good milling efficiency and no reaction with the metal powder. A further advantage, beside better dispersate distribution as against dry milling, is that the slurry can be discharged through a bottom valve in the Attritor without contact with air. The high temperature glowing connected with distilling off the liquid causes a sintering together of the powder. Thus the "submicron" powder after milling is no longer pyrophoric. Furthermore, a higher filling density and a better flowing property result. /23

As the experiments on compacting the powder have shown, hot compacting processes have to be used to obtain completely dense dispersion-hardened alloys. For that, both hot extrusion and isostatic hot compression are suitable. One or the other process can be used, according to the form of the parts to be compacted. Here extrusion serves especially for rod-shaped semi-finished

products, while isostatic hot pressing is particularly suitable for production of shaped parts. In either process attention has to be paid to a minimum filling-capacity of 50% for powder to be encapsulated, otherwise, when pressed, the capsules become too greatly deformed or can tear. In the use of wet milled powders with low filling density, a pre-compaction by pressing in a mold or by isostatic cold-pressing is expedient, however, for a higher degree of accuracy of the shaped parts, by isostatic hot-pressing.

Optimum pressing conditions for isostatic hot-pressing were determined as 1200°C/ h hr at 1000 bar. But falling below the pressing temperature and pressure can cause formation of micropores, which, in the subsequent homogenization heat, grow rapidly and clearly impair the mechanical properties. On the other hand indeed - as is known from works on the subject (21, 22) - by increasing the pressing temperature and pressure, a grain coarsening is achieved (Figure 8), which is absolutely necessary to attain good high-temperature strength.

Prolonged annealing at high temperatures near the alloy's melting point is another method for forming coarse grain. This can be a disadvantage due to coagulation of dispersate particles at high temperature. Moreover, it still is /24 not settled that alloys prepared by high-temperature annealing can be recrystallized at all. In the case of the alloy IN 853 (12, 16), it apparently is possible.

As numerous publications show (23 - 30), by thermomechanical treatment, i.e. by designed annealing and working one after another, recrystallization is attained. One should strive to form elongated grains with a high length/diameter ratio (L/D ratio), which lie parallel to the subsequent direction of stress (31 - 34). An explanation of this is that grain boundary glide can be regarded as the dominant working mechanism at high temperatures. Nevertheless if the majority of the grain boundaries lie parallel to the direction of stress, the shear tension acting on the grain boundaries is low, resulting in a better glide behavior of the alloys.

The emphasis of previous works was on the production of alloying powder and its compaction. A further decisive step is the regulation of a suitable structural condition. If the latter processes for grain enlargement are applied to the alloys produced, the high-temperature stability expected for dispersion-hardened alloys may be achieved. Such experiments are in progress now.

6. Summary

The investigations in developing of dispersion-hardened, temperable nickel alloys have shown that the production of such alloys by powder metallurgy is possible through mixing and milling of elemental metal powders or prealloys together with the dispersate in an agitator ball mill (Attritor). Two procedures are used for it, dry and wet milling, the advantages and disadvantages of which are discussed in detail. The charging of coarse-grained intermetallic phases /25 into the Attritor is shown to be advantageous, since they are easily milled to small dimensions and, as fine-grained powder, have a lower oxygen content.

Numerous experiments for compacting the powder were carried out in an isostatic hot-press. As optimal pressing conditions, 1200°C/1 hr/1000 bar were determined. The metallographic and electron microscopic studies showed a good dispersate distribution. By powder metallurgical production a fine-grained structure was obtained, which behaved superplastic at high temperatures.

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Translated for the National Aeronautics and Space Administration under contract No. NASW-2485 by Techtran Corporation, P. O. Box 729, Glen Burnie, Maryland, 21061, translator: William A. Peabody, Ph.D.